

PATENT APPLICATION Mo-6801 LeA 34,953

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF	)
CHRISTOPH GÜRTLER ET AL	) EXAMINING GROUP NO: 1621
SERIAL NUMBER: 10/085,514	) EXAMINER: K. J. PUTTLITZ
FILED: FEBRUARY 28, 2002	) )
TITLE: POLYISOCYANATES CONTAINING ACYLUREA GROUPS, A PROCES FOR THEIR PRODUCTION AND THEIR USE	

## **DECLARATION OF CHRISTOPH GÜRTLER**

I, Christoph Gürtler, residing in <u>Colput</u> , Germany, hereby declare as
follows:
1) I studied chemistry at the University of <u>Roum</u> from <u>1987</u> to <u>1993</u>
2) I received the degree of doctor at the University of in the year
3) I have been employed by Bayer AG, D-51368 Leverkusen, Germany, since
Polyworthane
4) I have been working in the research field of coatings since
1999 and I am the inventor of the subject matter of the above-identified
application; and

## Examples of yellowing carried out in the presence of various catalysts

604.8 g of hexamethylene diisocyanate and 11 mg of catalyst were initially introduced into a 1 l three-necked flask. 21.92 g of adipic acid and 9.41 g of azelaic acid were added to this mixture. After the addition was complete the temperature was adjusted to 120°C. Heating was carried out over the periods listed in the table below. Then the mixture was allowed to cool. The NCO value of the solution was about 43.4%. The reaction solution was subjected to thin-layer distillation (temperature: 130°C, pressure: 1.5 x 10<sup>-2</sup> bar). 140 to 150 g of a product were obtained with the colour index indicated in the Table in [Apha] and a viscosity which in all cases was within a range of 2000 to 2400 mPas. The yield was between 22.5 and 23.5%, based on the isocyanate. The content of monomeric hexamethylene diisocyanate in the thin-layer-distilled product was about 0.1%.

These comparative experiments were carried out without the addition of BHT or Tinuvin 770 which are used to reduce coloration when the material is exposed to higher temperatures. So no adulteration took place.

The choice of the catalyst has an effect on colouring during the reaction. Thus magnesium, calcium and lathanoid salts – the catalysts used according to the invention - produce only slightly yellow products, whereas the catalysts of Brahm produce products which are more yellow.

Catalyst	Colour index after thin-layer distillation	Reaction time in hours
Magnesium		
perchlorate	65	12
Calcium acetate	70	11
Ytterbium triflate	70	11
Triethylamine	120	15
Iron II chloride	250	11
Tin II octate	110	15
Dibutyl tin oxide	110	15
Phosphoric acid	150	14
	Magnesium perchlorate Calcium acetate Ytterbium triflate Triethylamine Iron II chloride Tin II octate Dibutyl tin oxide	thin-layer distillation  Magnesium perchlorate 65 Calcium acetate 70 Ytterbium triflate 70 Triethylamine 120 Iron II chloride 250 Tin II octate 110 Dibutyl tin oxide 110

Only products with a colour index of below 100 (Apha) can be used commercially.

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I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Leverkusen, Germany, this 16 day of November, 2005.

BAYER MATERIAL SCIENCE AG

CHRISTOPH GÜRTLER